IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of

MOK ET AL

Serial No. 09/361,626

Filed: July 27, 1999

Group Art Unit: 1724

Examiner: I. Cintins

For: PROCESS FOR REMOVING ALUMINUM SPECIES FROM ALKALI METAL HALIDE BRINE SOLUTIONS

March 7, 2000

RESPONSE TO OFFICE ACTION

Honorable Commissioner of
Patents and Trademarks
Washington, D.C. 20231

Sir:

In response to the Office Action of December 7, 2000, reconsideration of this application is requested.

The Examiner has rejected the applicants' claims 1-7, 9 and 10 under Section 103(a) as unpatentable over NAGY. In rejecting the claims, the Examiner notes that Nagy discloses a process for removing trace amounts of aluminum from a sodium chloride brine by treating the brine with magnesium chloride to produce a magnesium concentration of at least about 5 ppm in the brine, and subsequently adding sodium hydroxide to this brine to provide an alkalinity concentration of 0.15 g/L in this brine, thereby forming a magnesium aluminum hydroxide precipitate.

The Examiner then acknowledges that the reference does not disclose the applicants' recited magnesium to aluminum molar ratio, and the magnesium concentration in the brine

(i.e. less than 5 ppm). Nevertheless, the Examiner concludes that the "exact magnesium to aluminum molar ratio in the reference process is not seen to materially affect the overall results of this process, or to produce any new and unexpected result; and is therefore deemed to be an obvious matter of choice, which is insufficient to patentably distinguish to the claims. Furthermore, it would have been obvious to one of ordinary skill in the art at the time the invention was made to employ a brine having a magnesium concentration slightly lower than that disclosed (e.g. 4.99 ppm) in the reference, since such a slightly lower magnesium concentration is also not seen to materially affect the overall results of the reference process, or to produce any new and unexpected result; and is therefore also deemed to be an obvious matter of choice, which is insufficient to patentably distinguish the claims."

In response to the Examiner's position, the applicants respectfully submit that the Mg to Al molar ratio and Mg concentration called for in the claims, together with sufficient alkali metal hydroxide to provide the excess alkalinity called for by the applicants (0.1 to 0.3 g/L) provide unexpected results which support patentability of the claimed matter.

The unobviousness of the applicants' method over Nagy is shown by the attached experimental results and graphs wherein conditions representative of the applicants' method and Nagy's method are compared. The attachments include the results of three experiments and a further experiment representative of Nagy. The results of these experiments are summarized in the "Conclusion" section. The tabulated results of the experiments show how the efficiency of Al removal varies with Mg content, Mg/Al ratio and alkali concentration (NaOH).

The "Conclusion" section (pages 3-4 of the attachment) brings out the importance of observing the specific conditions called for in the applicants' claims. The significance of those conditions is not in any sense disclosed or suggested by Nagy (Diamond Shamrock).

The attached charts (pages 5 and 6 of the attachment) graphically illustrate the results obtained in the applicants' method and Nagy's method. The applicants' result clearly could not have been predicted from the Nagy results.

Favorable reconsideration of the Section 103(a) rejection with allowance of the claims is requested in view of the foregoing and attached evidence.

Respectfully submitted,

PILLSBURY WINTHROP, LLP

By _____

Paul N. Kokulis
Reg. No. 16,773
Phone: 202.861.3503

PNK:mll
1100 New York Avenue, N.W.
9th Floor, East Tower
Washington, D.C. 20005-3918
Phone: (202) 861-3000

Optimization of Aluminum Removal by Magnesium Chloride Addition

The following results have been tabulated to illustrate the effects of Aluminum removal using Magnesium Chloride at various operating conditions.

Temperature: 50°C Brine: Ion-Exchanged Quality
Magnesium Chloride & NaOH: Reagent Grade Quality

Experiment #1

Al (ppm)	Mg (ppm)	Mg/Al Molar Ratio	NaOH (g/L)	Residence Time (Minutes) 0 Residual Al (ppm)	0 Removal Efficiency (%)	15 Residual Al (ppm)	15 Removal Efficiency (%)	30 Residual Al (ppm)	30 Removal Efficiency (%)
0.1	0.5	5.6	0.1	0.064	36	0.034	66	0.028	72
0.1	0.5	5.6	0.2	0.046	54	0.044	56	0.039	61
0.1	0.5	5.6	0.5	0.055	45	0.055	45	0.049	51
0.2	0.5	2.8	0.1	0.130	41	0.119	46	0.115	48
0.2	0.5	2.8	0.2	0.139	37	0.130	41	0.129	41
0.2	0.5	2.8	0.5	0.173	21	0.139	37	0.139	37
0.5	0.5	1.1	0.1	0.390	22	0.390	22	0.380	24
0.5	0.5	1.1	0.2	0.430	14	0.430	14	0.440	12
0.5	0.5	1.1	0.5	0.440	12	0.410	18	0.420	16

Experiment #2

Residence Time (Minutes)

Al (ppm)	Mg (ppm)	Mg/Al Molar Ratio	NaOH (g/L)	0 Residual Al (ppm)	0 Removal Efficiency (%)	15 Residual Al (ppm)	15 Removal Efficiency (%)	30 Residual Al (ppm)	30 Removal Efficiency (%)
0.1	1.0	11.1	0.1	0.009	91	0.009	91	0.009	91
0.1	1.0	11.1	0.2	0.013	87	0.010	90	0.008	92
0.1	1.0	11.1	0.5	0.028	72	0.018	82	0.014	86
0.2	1.0	5.6	0.1	0.065	68	0.070	65	0.060	70
0.2	1.0	5.6	0.2	0.103	49	0.094	53	0.090	55
0.2	1.0	5.6	0.5	0.109	46	0.097	52	0.094	53
0.5	1.0	2.2	0.1	0.268	46	0.251	50	0.252	50
0.5	1.0	2.2	0.2	0.332	34	0.309	38	0.327	35
0.5	1.0	2.2	0.5	0.362	28	0.363	27	0.359	28

Experiment #3

Residence Time (Minutes)

Al (ppm)	Mg (ppm)	Mg/Al Molar Ratio	NaOH (g/L)	0 Residual Al (ppm)	0 Removal Efficiency (%)	15 Residual Al (ppm)	15 Removal Efficiency (%)	30 Residual Al (ppm)	30 Removal Efficiency (%)
0.1	2.0	22.2	0.1	0.012	88	0.015	85	0.011	89
0.1	2.0	22.2	0.2	0.011	89	0.012	88	0.013	87
0.1	2.0	22.2	0.5	0.010	90	0.009	91	0.012	88
0.2	2.0	11.1	0.1	0.020	90	0.014	93	0.014	93
0.2	2.0	11.1	0.2	0.033	84	0.017	92	0.017	92
0.2	2.0	11.1	0.5	0.055	73	0.023	89	0.015	93
0.5	2.0	4.4	0.1	0.175	65	0.162	68	0.159	68
0.5	2.0	4.4	0.2	0.194	61	0.209	58	0.206	59
0.5	2.0	4.4	0.5	0.268	46	0.252	50	0.240	52

Diamond Shamrock Corporation (Zoltan Nagy) U.S. Patent No. 4,073,706

Temperature : 66°C

Al (ppm)	Mg (ppm)	Mg/Al Molar Ratio	NaOH (g/L)	20 Minutes of Residence Time Residual Al (ppm)	Removal Efficiency (%)
1.0	2.5	2.8	0.1	1.0	0
1.0	15	16.7	0.5	0.14	86
1.0	62	68.8	0.5	0.26	74
1.0	122	135.5	0.5	0.16	84

Al (ppm)	Mg (ppm)	Mg/Al Molar Ratio	NaOH (g/L)	60 Minutes of Residence Time Residual Al (ppm)	Removal Efficiency (%)
1.0	15	16.7	0.5	0.3	70

Al (ppm)	Mg (ppm)	Mg/Al Molar Ratio	NaOH (g/L)	180 Minutes of Residence Time Residual Al (ppm)	Removal Efficiency (%)
1.0	68	75.5	0.9	0.08	92
1.0	146	162.1	0.9	0.06	94
1.0	122	135.5	0.5	0.04	96

Conclusion:

Aluminum removal efficiency of 90% has been successfully demonstrated when only 1 ppm and 2 ppm of Mg in the form of MgCl2 was added to a brine solution containing 0.1 and 0.2 ppm Al with 0.1 to 0.2 g/L excess NaOH. This translates to a Mg to Al molar ratio of 11 to 1. Refer to Experiment #2 & #3 data tables. Furthermore, there is
also a delicate balance existing between the excess alkalinity level and the Magnesium concentration in the brine solution.

Once all the Magnesium ions have been consumed to forming the gelatinous hydroxide precipitate (amphoteric nature), the excess hydroxide anions tend to re-solubilize the complex precipitate thus releasing the Aluminum impurities back into solution. This effect is clearly indicated by the lower Aluminum removal efficiency at the higher excess alkalinity concentrations. Refer to Experiment #1, #2 & #3 data tables.

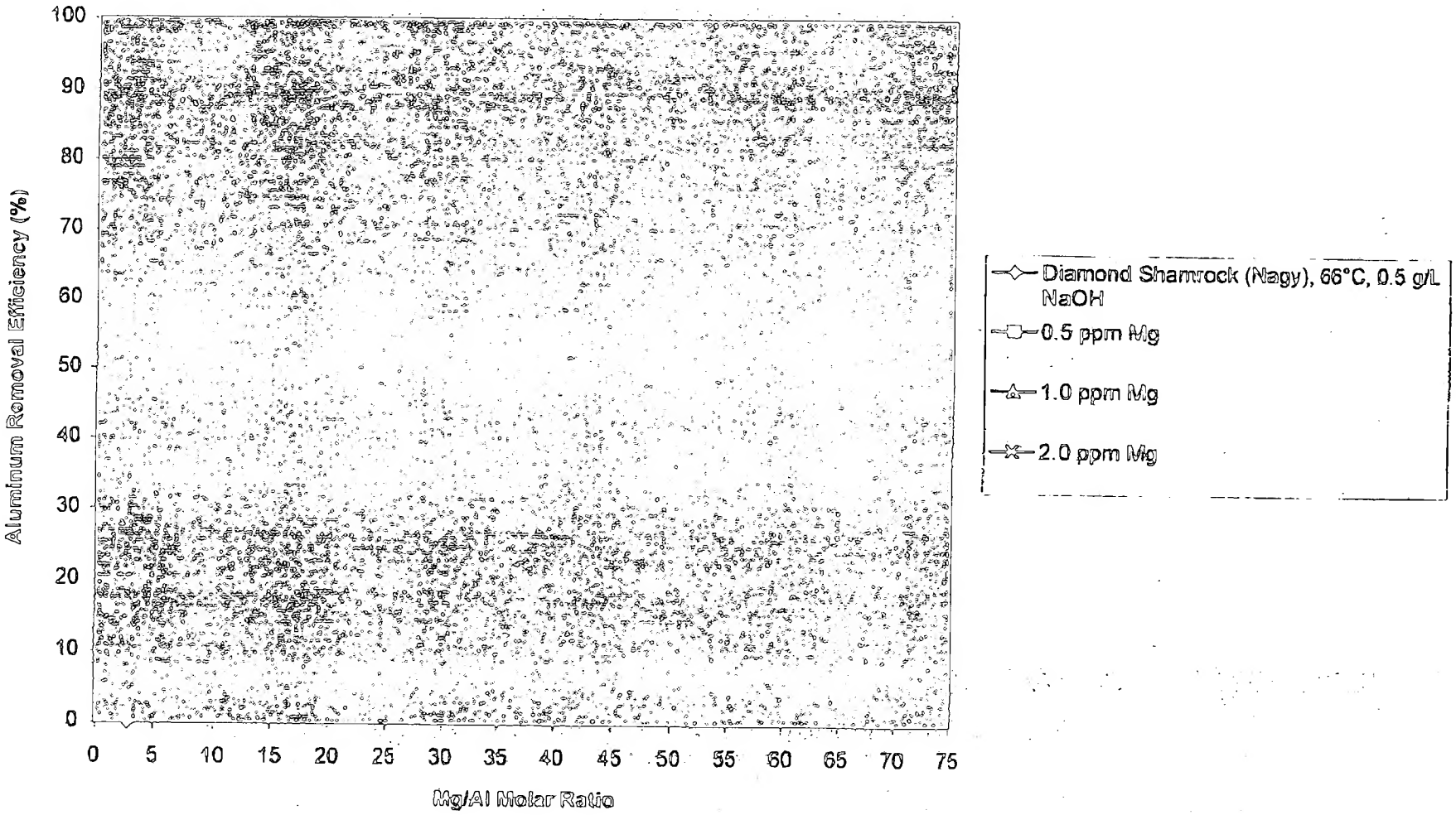
The reaction kinetics of this complex precipitation mechanism is instantaneous where upon only traces of Magnesium, 0.5 to 2.0 ppm, in the presence of 0.1 to 0.2 g/L NaOH, the removal of Aluminum is almost immediate and increase in residence time has not significantly improved on the overall removal efficiency.

The Diamond Shamrock Patent clearly cites that Magnesium level of less than 5 ppm concentration have been found to be ineffective in removing many of the undesirable metallic impurities including Aluminum, as indicated in its example where zero Aluminum removal efficiency was attained in a brine solution containing 1 ppm Al with using 2.5 ppm Mg and 0.1 g/L NaOH excess alkalinity after 20 minutes of reaction time. The working ratio of Mg to Al for this particular example translates to 2.8 to 1. When compared to a similar working Mg/Al ratio in our claims but using only 0.5 to 1.0 ppm Mg, a 50% Aluminum removal efficiency was achieved. Refer to Experiment #1 &#2 data tables and Chart #1 & #2.

This Aluminum removal process using low levels of Magnesium concentration has been successfully operated at two separate chloralkali plants in the United States. The Chloromone Co. in Delaware City was the first plant to fully utilize the benefits of this technology. It was commissioned back in January 1999 with maximum capacity of 50 stpd of chlorine achieved after one month of operation. The second plant which has adopted the identical treatment process was Odyssey Manufacturing Co. located in Tampa, Florida. The chloralkali facility was commissioned back in March 2000 with completion of performance test achieved after only two weeks of operation.

(5)

Chart 1 - Comparison of Aluminum Removal Efficiency (%) at Various Mg/Al Molar Ratio with Low Magnesium Concentration
Temperature at 50 to 66°C; Excess Alkalinity Level at 0.5 g/L NaOH



(6)

Chart 2 - Comparison of Aluminum Removal Efficiency (%) at Various Mg/Al Ratio with Low Magnesium Concentration
Temperature 50°C; Excess Alkalinity Level at 0.1 g/L NaOH